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Cement Research in the U.S.S.R.

POZZOLANA CEMENTS

IN view of the large amount of concrete work carried out in the U.S.S.R., the Government of that country set up a committee which for six years (from 1929 to 1935) has studied the effect of sea water, sulphates and other aggressive solutions on concrete, with particular reference to the use of pozzolana as a preventive. As a result of this research there is now in the U.S.S.R. a considerable production of pozzolanic cement containing admixtures of volcanic (Karadag trass) and of sedimentary (diatomite, etc.) origin. At other plants an addition of from 10 to 15 per cent. of pozzolana is made to the cement clinker before grinding; in these cases the product is still called "Portland cement." The reasons for this more extended use of pozzolanic Portland cement are said to be: (1) improved resistance to corrosion, (2) improved durability in hydraulic structures, (3) lower cost of production.

Abstracts of some of the papers published by the committee are given in the following.

Effect of Sulphates on Ordinary and Pozzolana Portland Cements.

By A. A. BAIKOV and A. S. TUMAREV.

The deleterious effect of natural water upon concrete is caused by various substances dissolved in the water, particularly magnesium salts and calcium and sodium sulphates. Excluding aluminous cement, pozzolana Portland cement is considered to be the most suitable for hydraulic structures. Mortars and concretes made with these cements have higher resistance to the action of natural water, and greater endurance in the presence of sulphates, in comparison with ordinary Portland cements.

In this investigation a series of tests was made on specimens of neat cement and of 1:3 mortar. After 28-day tensile strength tests the specimens were immersed in calcium and sodium sulphate solutions, the initial composition of the solutions being restored from time to time. The specimens were under

continuous observation, and records were made of all variations in shape, decomposition, cracking, efflorescence, etc.

The subjects of this investigation were sodium and calcium sulphates of three different concentrations, namely, (*a*) sodium sulphate, 10 per cent., 1 per cent., and 0·1 per cent.; (*b*) calcium sulphate, saturated (0·2 per cent.), half saturated (0·1 per cent.) and quarter saturated (0·05 per cent.). Four different Portland cements differing in CaO content and in strength (standard test), one slag cement, and two pozzolana Portland cements (one with Karadag trass and the second with Briansk diatomite) were investigated. After four years the following results were obtained:

(1) All cements except the pozzolana Portland cements showed more or less pronounced deterioration under the attack of Na_2SO_4 and $CaSO_4$ solutions; the pozzolana Portland cement, both neat cement and mortar specimens, remained intact.

(2) The destructive effect was more pronounced in the 1 : 3 mortar specimens in strong solutions of Na_2SO_4 (10 per cent.) and $CaSO_4$ (0·2 per cent.), the rate of disintegration being the highest in the sodium sulphate solution.

(3) The less the concentration of the solutions the slower the rate of disintegration and the later the signs of destruction appear.

There is no relation between the strength of cement mortars and their resistance to mineral agents. Thus the selection of cement for hydraulic structures should not be based on their strength only.

Accelerated Tests for the Resistance of Cements to Salt Waters.

By V. V. KIND.

Some investigators who have studied the resistance of cement to salt solutions recommend the use of highly concentrated solutions (10 to 15 per cent.) and the curing of the samples at high temperature in order to speed up the disintegration of the samples. This method is open to question as it means that the cements would be tested under conditions completely different from the actual conditions of hydraulic structures. The results of tests show that a higher concentration not only causes a higher rate of disintegration of the samples but often changes the character of the action of the solution upon the cements so that the relative (comparative) resistance of the cements is very different from that in weak solutions.

The tests described here were made on specimens of very small size (cubes of 1·41 cm. side) and bars measuring 1 cm. by 1 cm. by 3 cm., made by means of the apparatus of Professor Kühl. The specimens are made of a 1 : 3·5 cement to sand mortar with river quartz sand, using particles passing a sieve with 144 meshes per square centimetre and retained on a sieve with 256 or 225 meshes per square centimetre. Owing to the round shape of the grains and their uniform size this mortar was porous enough to secure the penetration of water into the specimens. The specimens were cured under moist conditions for one day and in water for 14 days, and were then placed in the required solution or mineral

water. Some of the specimens were placed in fresh water (tap water). After certain periods of immersion in the solution some of the specimens were tested ; the cubes for compressive strength, and the bars for tensile strength.

The analysis of the strength variation of the bars and cubes allows some conclusions to be drawn on the resistance of cements to the given solution. The results obtained with each solution should be compared with those obtained for the specimens stored in water. To meet the requirements of many hydraulic construction works these investigations were carried out and furnished the necessary data on the comparative resistance of various cements to mineral waters of various compositions. In spite of the specimens having often been cured in natural water, and regardless of the presence of some small quantities of salt in it, definite results were obtained in 3 to 6 months.

Effect of Sulphate Solutions on Various Cements.

By V. V. KIND.

The object of this investigation was the study of the resistance of various cements to concentrated (10 per cent.) sodium sulphate and magnesium sulphate solutions and also to saturated calcium sulphate solution. This investigation covered three brands of Portland cements with various hydraulic (pozzolanic) additions and one slag cement. The pozzolanic admixtures used were trass, diatomite, and Si-stoff. The determination of the relative resistance of these cements was based on the accelerated tests developed by the author. The results show that of all the cements investigated the least resistance to saturated calcium sulphate solutions and to 10 per cent. sodium sulphate solution was exhibited by the Portland cements. After a short time the strength of the Portland cement specimens was greatly affected and they were soon completely disintegrated. The pozzolana and slag cements withstood the attack of calcium sulphate and sodium sulphate solutions very well. No signs of disintegration were observed.

The behaviour of the cements in 10 per cent. magnesium sulphate was completely different. The pozzolana and slag Portland cements disintegrated in this solution rather rapidly, whereas Portland cement showed a much slower reduction in strength.

Effect of Dilute Sulphate Solutions on Various Cements.

By V. V. KIND.

The majority of workers who have investigated the chemical resistance of cement to salts used for their tests strong sulphate solutions, the concentration varying from 5 per cent. to 15 per cent. In this case the disintegration of the cement proceeds under conditions completely different from the actual conditions of service of hydraulic structures.

For this investigation the following dilute solutions were taken, the concentration being approximately the same as of natural mineral water containing these salts: (1) $CaSO_4$; (2) Na_2SO_4; (3) $MgSO_4$; (4) $CaSO_4 + Na_2SO_4$;

(5) $CaSO_4 + MgSO_4$; (6) $Na_2SO_4 + MgSO_4$; (7) $CaSO_4 + Na_2SO_4 + MgSO_4$. The concentration of all solutions was 0·03 (for the solutions containing several salts the total concentration is estimated). Two Portland cements, three pozzolana (blended) cements, and one slag cement were tested in these solutions.

A method of accelerated tests developed in the Building Materials Laboratory of the Scientific Research Institute for Hydraulic Engineering was used. Small specimens (cubes and bars) made of 1 : 3·5 mortar were tested. The resistance of the cements was determined by the strength variation of the specimens stored in various solutions. Besides these weak solutions, 10 per cent. solutions of Na_2SO_4 and $MgSO_4$ were taken for comparison. The tests show that the influence of weak sulphate solutions, independently of their composition, is practically the same. The Portland cements disintegrate in these solutions rather rapidly (the rate of disintegration being nearly the same in all solutions), whereas the specimens made of pozzolana and slag cements show no sign of disintegration whatever and, moreover, the hardening process proceeds even better than in fresh water. The comparative chemical resistance of cements in 10 per cent. Na_2SO_4 solution was found to be nearly the same as in dilute solutions. Contrary results were obtained in the 10 per cent. $MgSO_4$ solution; the blended cements disintegrated rather rapidly, whereas the disintegration of Portland cements was much slower.

Thus the chemical action of $MgSO_4$ on cement changes greatly with the increase of its concentration, and consequently concentrated $MgSO_4$ solutions should not be used for laboratory investigations.

Resistance of Cement to Fresh Water.

By I. D. SAPOROJEZ.

The object of this work was to examine methods of determining resistance of cement to fresh water. The following were studied: (1) liberation of lime from hydrated cement; (2) leaching of lime from set cement powder when shaken with water; (3) diffusion of lime from cement mortar samples immersed in water under heating; (4) leaching of lime from cement mortar samples under the filtration of water. Most attention was given to a comparison of the water-resistant properties of Portland cement and pozzolana cements.

The following conclusions were reached: (1) The water-resistant properties of a cement depend on its solubility and on its inherent property to impart more or less density to the mortar and concrete. Both of these properties should be considered in the estimation of resistance to water. (2) The data obtained from the shaking of cement powder with water are insufficient for the evaluation of the resistance of cement to water. This method may serve only for the evaluation of the solubility of the cement. (3) The most reliable method for the determination of the water-resistance of cements is the filtration of water through cement mortars. This is the method developed and recommended. (4) The values of the water-resistance of cements are expressed by the amount of leached (washed) lime, as a function of time (lime leaching curves) or as the average rate of leaching. (5) The water-resistance of pozzolana cements is many times higher than that of

Portland cement, which is attributed to the lower solubility and the higher density of pozzolana cement mortar. (6) The water-resistance of pozzolana cement is directly proportional to the reactivity of the hydraulic admixture used for the blend.

Shrinkage of Concrete.
By GLUJGE.

The main object of this work was the comparison of volume change in Portland cements, pozzolana Portland cement, and slag Portland cement. The results of observations at the age of 360 days may be summarised as follows: High-strength Portland cements (of the Novorossyisk and Leningrad cement plants) showed the least volume change under air curing. The shrinkage under moist curing (in damp sawdust) and combined curing (6 days in moist sawdust and 12 days in air) were less in all pozzolana and slag Portland cements than in Portland cements.

Effect of Hydraulic Admixtures on the Bond between Mortar and Steel.
By L. S. KOGAN.

This paper presents data on the bond properties of (*a*) Portland cement without admixtures, (*b*) Blended cements with additions, their percentage being limited to that admitted for Portland cement without changing its name, and (*c*) Pozzolana Portland cements. The investigation covered a series of cements blended with the following admixtures in various proportions: Karadag trass, Briansk diatomite, Si-stoff, Nalchik volcanic ash, ground quartz sand, and others. The total number of determinations was 30,000. It was fully established that the absolute adhesive power decreases with the increase of the percentage of the admixture. In this case, however, a corresponding loss of compressive strength is observed. The results showed that pozzolana Portland cements suitable for the production of mortars and concretes may also be used for reinforced concrete.

Effect of High Temperature on Cements.
By C. M. RUSCHUK.

At temperatures up to 500 to 600 deg. C. decomposition is mainly due to the dehydration of calcium hydroxide with subsequent secondary hydration of the calcium oxide through the absorption of air moisture or other contact with water. Since calcium hydroxide readily combines with the active silica in the hydraulic additions, pozzolana Portland cements should be considered to possess more fire-resistant properties.

An investigation involving physico-chemical and strength tests of set cements under temperatures ranging up to 900 deg. C. was carried out.

The results of these tests may be briefly summarised as follows:

The tests on pozzolana cements with Si-stoff (alumina production residues), slag cement, and aluminous cement showed the absence of calcium hydroxide $Ca(OH)_2$.

The contraction of all cements began over the temperature range 200 to 355 deg. C., the amount of contraction being different for different cements.

Pozzolana cements with Si-stoff, diatomite, and trass had the greatest contraction.

The heat evolution at secondary hydration was quite different for different cements. The more complete the dehydration of the calcium hydroxide the higher the heat of hydration.

Pozzolana cement was found to have a lower heat evolution at temperatures over 800 deg. C., which is evidently due to the process of combining the liberated dehydrated $Ca(OH)_2$.

Compressive strength tests showed that at the age of three months pozzolana Portland cements had greater resistance to temperatures up to 600 to 900 deg. C. compared with Portland cement.

A Portland cement sample exposed to air after having been heated to 900 deg. C. disintegrated, whereas pozzolana cement preserved a great part of its initial strength.

Pozzolana Portland Cements in Mass Concrete.

By W. N. YOUNG.

Cement used for mass concrete should be a low-heat cement possessing sufficient resistance to the leaching action of fresh water and to the aggressive action of natural salt water. A pozzolana Portland cement, consisting of Portland cement ground with a proper proportion of highly siliceous hydraulic admixture, meets these requirements best. Concrete made of this cement has sufficient strength and impermeability. The leaching of lime from set pozzolana Portland cement proceeds at a much slower rate than from Portland cement without admixtures.

The rate of lime-leaching in powdered set cement samples, with particle size under 0·1 mm., is lower from the beginning than in ordinary Portland cement. In the course of further hardening of pozzolana cement the lime-leaching proceeds at a slower rate, which steadily decreases due to the combination of the hydrated lime with the hydraulic admixture and the resulting formation of more resistant hydrosilicates of calcium, whereas in ordinary Portland cement this rate increases with age in further hydration and the increase of free hydrated lime. The formation of calcium hydrosilicate due to the reaction between the silica of the hydraulic admixture and the liberated lime is a slow process so that a considerable part of the free lime is combined by the end of a month or so. From this it may be concluded that the heat evolution per unit of weight of pozzolana Portland cement during the hardening period is less than that of the same Portland cement without admixtures. Subsequent calorimetric tests showed that at early ages (4 and 7 days) the expected reduction is to a certain extent proportional to the percentage of the addition (diatomite), but at later ages (28 days and more) the amount of heat evolved by pozzolana Portland cement approaches that of Portland cement without the admixture. The author finds the explanation of this fact in the higher water-cement ratio and consequently the more complete hydration of the Portland cement particles contained in pozzolana cement. A special cement for hydraulic structures is now produced by grinding 60 to 70 per

cent. (by weight) of clinker (with 2 per cent. of gypsum) with 20 to 25 per cent. of dry diatomite and 10 to 15 per cent. of quartz sand.

High-Strength (Rapid-Hardening) Pozzolana and Slag Portland Cements.

By V. A KIND, S. D. OKOROKOV, and Z. V. FROLOV.

The rate of setting and hardening of pozzolana and slag Portland cements is slower than that of the Portland cement used for the blend. These cements consequently show low initial strength values and gain the strength of the Portland cement only after time. To eliminate this disadvantage an investigation was carried out to determine the influence of the chemical composition of the Portland cement clinker used for the blend on the properties of the cements. This investigation involved the burning in the laboratory of various clinkers of predetermined chemical composition, i.e. different saturation factors (lime saturation), silica ratios and alumina/iron oxide ratios. These clinkers were used for the preparation of pozzolana and slag Portland cements with different hydraulic admixtures (Karadag trass, Nalchik ash, Briansk diatomite, Kurinsk diatomite, Si-stoff, granulated basic blastfurnace slag from three metallurgical plants), and with different percentages of these admixtures in the blended cement.

The results show that the rate of strength development in pozzolana and slag cements depends to a great extent on the chemical composition of the clinker used for the blend. To produce rapid-hardening pozzolana and slag Portland cements, the following chemical composition is necessary: (1) A high saturation factor varying between 1·00 and 1·05; (2) A high silica ratio ranging from 4·00 to 5·00; and (3) an alumina/iron oxide ratio not less than 2·45. Clinkers of this chemical composition produce high quality cements if they are burnt at the temperature required for their normal sintering. The resulting pozzolana and slag Portland cements have a greater rate of strength development at early ages than if the clinkers are insufficiently burned. The percentage of the hydraulic admixture added to the clinker is nearly the same for rapid-hardening pozzolana and slag Portland cements as for ordinary pozzolana and slag cements. The need to increase this percentage may arise only in particular cases.

Hydraulic Properties of Acidic Blastfurnace Slag.

By V. A. KIND and M. S. KUROZAPOV.

While basic granulated blastfurnace slag has been used for cement production the possibility of the use of acidic slags has only recently been established. This was mainly due to the declaration of well-known authorities that acidic slags with a $\frac{CaO + MgO}{SiO_2 + Al_2O_3}$ ratio less than unity cannot be used for the production of cement.

The authors investigated such acidic slags from three metallurgic mills in the Urals which smelt cast iron using charcoal for fuel. These slags have the following $\frac{CaO + MgO}{SiO_2 + Al_2O_3}$ ratios: (*a*) 0·70; (*b*) 0·82; (*c*) 0·58. Tests of cements prepared of ground slag with slaked lime show that rapidly-cooled acidic slag

possesses high reactivity. The rate of strength development under water-curing conditions is much higher than under air curing. In the latter case, a clearly pronounced reduction in strength is observed in the period between six months and one year. In other words, the behaviour of these cements is similar to that of lime-pozzolana cements.

Tests of pozzolana Portland cements with the addition of acidic blastfurnace slag show that at the age of three months certain blends of these cements reach higher strength values than the Portland cement used for the blend. But at shorter dates (7 and 28 days) the strength of the blended cement is in most cases (but not always) lower than that of Portland cement. In other words they behave as typical pozzolana cements. Pozzolana Portland cements with acidic blastfurnace slag show higher strength when air cured as compared with water curing.

Revision of the German Standard Specifications for Portland, Slag, and Blast-Furnace Cements.

THE German Ministries for Transport and Public Works have, by a by-law of June 20, 1939, altered the German standard specifications for cement. The amendment refers mainly to the required ultimate strengths, as shown in the tables on the facing page, showing the requirements of this by-law and of the last specification dated 1932.

It is seen that the requirements for the compression strength of normal Portland cement have been increased, whereas the tension and the compression strengths for high-grade cement are unchanged.

Instead of pure tension tests, in future the tensile stress is to be found by bending tests. For normal and high-grade cements the tests for tension due to bending shall show the following minimum results:

(*a*) Normal cement: After 7 days' storage in water, 356 lb. per sq. in.; after 28 days' storage in water, 712 lb. per sq. in.

(*b*) High-grade cement: After 7 days' storage in water, 356 lb. per sq. in.; after 28 days' storage in water, 783 lb. per sq. in.

The tests for tensile stress due to bending after 28 days' combined storage may be omitted. The method of testing is to be in accordance with the German standard specifications (DIN 1165) "Arrangements for making and testing of prisms 4 × 4 × 16 cm. of soft-mixed mortar" and DIN 1166, "Preparation and testing of prisms of soft-mixed mortar."

The test after 28 days of combined storage (1 day moist air, 6 days water, 21 days air) and the standard compression test are in future to be omitted for normal and high-grade cements. Until June 30, 1940, when the new bending-tensile test comes into force, either the new test or the old tensile test may be used.

CHANGES IN GERMAN STANDARD SPECIFICATIONS FOR PORTLAND, SLAG, AND BLAST-FURNACE CEMENTS.

(*See facing page*).

STANDARD SPECIFICATION OF 1932.

Mortar strength after	3 days' storage in water	7 days' storage in water	28 days' combined storage	28 days' storage in water
		CEMENT		
Compression .. .. (in lb. per sq. in.)	—	2,560	4,978	3,911
Tension .. .. .. (in lb. per sq. in.)	—	256	427	356
		HIGH-GRADE CEMENT		
Compression .. .. (in lb. per sq. in.)	3,556	—	7,111	5,689
Tension .. .. .. (in lb. per sq. in.)	356	—	569	427

REVISION OF JUNE 20, 1939.

Mortar strength after	3 days' storage in water	7 days' storage in water	28 days' combined storage	28 days' storage in water
		CEMENT		
Compression .. .. (in lb. per sq. in.)	—	2,845	5,689	4,267
Tension .. .. .. (in lb. per sq. in.)	—	256	427	356
		HIGH-GRADE CEMENT		
Compression .. .. (in lb. per sq. in.)	3,556	—	7,111	5,689
Tension .. .. .. (in lb. per sq. in.)	356	—	569	427

Constitution of Portland Cement Clinker *

By R. H. BOGUE

In these calculations there is postulated no partial solidification of the liquid as glass—it is assumed either to solidify completely as glass from the temperature or condition given or to crystallise completely but independently (except in the first case where equilibrium crystallisation is assumed). Under these assumptions, even at complete independent crystallisation, the percentages of the calculated compounds are seen to differ from those computed at equilibrium crystallisation. A critical study of the basic data shows that, within the $Al_2O_3 : Fe_2O_3$ ratio range of 0·94 to 1·66, no change in composition results through the independent crystallisation of the liquid but, with $Al_2O_3 : Fe_2O_3$ ratios below 0·94, the $3CaO.SiO_2$

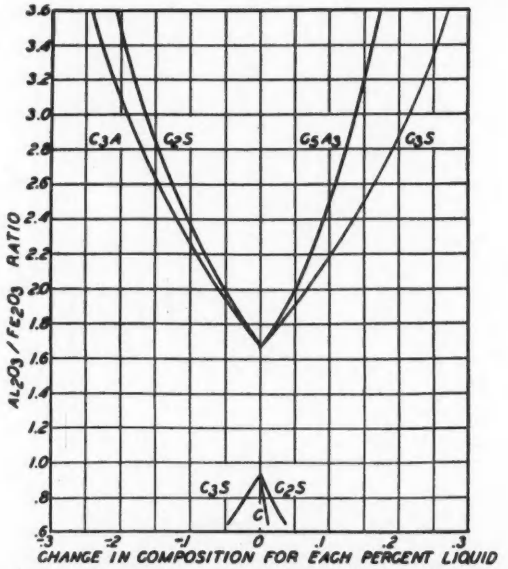

Fig. 3.—Change in Composition of System through Complete Crystallisation of Liquid independently from 1400°C.

is decreased and $2CaO . SiO_2$ increased (accompanied by the separation of free CaO), whereas with ratios above 1·66 the reverse obtains. Furthermore, the $3CaO . Al_2O_3$ is reduced at $Al_2O_3 : Fe_2O_3$ ratios above 1·66 and some Al_2O_3 separates out as $5CaO . 3Al_2O_3$. These changes in composition for each per cent. of liquid are shown graphically in *Fig.* 3, drawn from calculations by Dahl.

On the assumption that all of the liquid solidifies as glass, the iron and alumina compounds are absent from the crystalline phases provided the crystallisation occurs at some temperature above that of the point where the liquid composition

*Previous parts of this paper were published in this journal for October and November, 1939.

leaves the $3CaO \cdot SiO_2$ boundary surface with $3CaO \cdot Al_2O_3$ or $4CaO \cdot Al_2O_3 \cdot Fe_2O_3$. The same order of effect on the silicates is observed as in the condition of independent crystallisation.

Dahl[47] has extended the computations of Lea and Parker to include 7 types of composition wherein the course of crystallisation differs from that of every other type in the 4-component system under consideration. These 7 types are shown in *Fig.* 4 where the points E_p, $(T_1)_p$ and $(T_2)_p$ are projections of the points E, T_1 and T_2 (cf. Lea and Parker's Techn. Paper 16) from the $3CaO \cdot SiO_2$ point to the $2CaO \cdot SiO_2$-$3CaO \cdot Al_2O_3$-$4CaO \cdot Al_2O_3 \cdot Fe_2O_3$ plane.

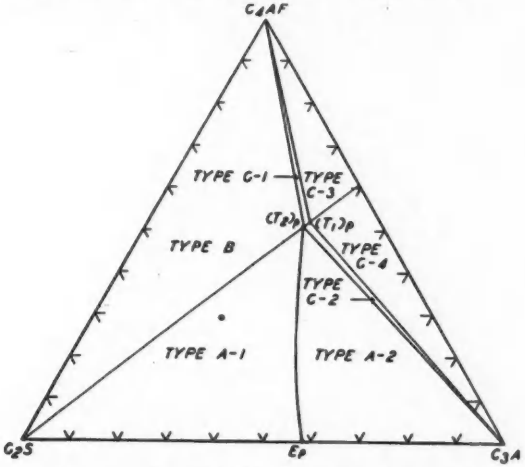

Fig. 4.—Diagram showing Composition Types

The course of crystallisation followed by the 7 different types of cement is as follows:

Type *A-1.* At 1,450 deg. and 1,400 deg. the liquid is on the $3CaO \cdot SiO_2$-$2CaO \cdot SiO_2$ surface. It reaches the quintuple line T_2E at a temperature which depends on the composition (equations for the calculation of which are given in Dahl's paper), and solidifies at T_2 at 1,338 deg.

A-2. Liquid passes from CaO-$3CaO \cdot SiO_2$ surface to T_1D, thence to $3CaO \cdot SiO_2$-$3CaO \cdot Al_2O_3$ surface, thence to T_2E and solidifies at T_2.

B. Liquid passes from $3CaO \cdot SiO_2$-$2CaO \cdot SiO_2$ surface to T_2W, thence to T_2.

C-1. Liquid passes from CaO-$3CaO \cdot SiO_2$ surface to T_1X, thence to $3CaO \cdot SiO_2$-$4CaO \cdot Al_2O_3 \cdot Fe_2O_3$ surface, thence to T_1T_2 and solidifies at T_2.

C-2. Liquid passes from CaO-$3CaO \cdot Al_2O_3$ surface to T_1D, thence to $3CaO \cdot SiO_2$-$3CaO \cdot Al_2O_3$ surface, thence to T_1T_2 and solidifies at T_2.

C-3. Liquid passes from CaO-$3CaO \cdot SiO_2$ surface to T_2X, thence to T_1, thence to T_1T_2 and solidifies at T_2.

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[47] DAHL, L. A. *Rock Products*, in press.

C-4. Liquid passes from $CaO\text{-}3CaO \cdot SiO_2$ surface to T_1D, thence to T_1, thence to T_1T_2 and solidifies at T_2.

The types *A-1*, *A-2*, *C-2* and *C-4* are included in Lea and Parker's type (a) and the types *B*, *C-1*, and *C-3* in their type (b). Types *C-3* and *C-4* represent compositions of low $2CaO \cdot SiO_2$ content in which free CaO is present at equilibrium at 1,341 deg. Types *A-2*, *C-1*, and *C-2* include compositions which may contain free CaO at equilibrium at slightly higher temperatures, the temperature depending on the $2CaO \cdot SiO_2 : 4CaO \cdot Al_2O_3 \cdot Fe_2O_3$ ratio for types *A-2* and *C-2* and on the $2CaO \cdot SiO_2 : 3CaO \cdot Al_2O_3$ ratio for type *C-1*.

Equations have been set up by Dahl whereby the composition of the mixtures in the 4-component system may be calculated at each of the critical points referred to, under the following conditions of cooling: (1) For all types of composition, where all of the liquid in equilibrium at the temperature cited has solidified as glass; (2) For all types of composition, where all of the liquid has solidified by independent crystallisation; (3) For type *A-1* and *B* compositions, where normal crystallisation is assumed to have proceeded to an estimated glass content; and (4) For type *A-1* composition, where crystallisation is assumed to have proceeded independently from 1,338 deg., 1,400 deg., and 1,450 deg. to an estimated glass content.

The magnesia may be included on the assumption that liquids on surfaces or at invariant points in the quaternary system $CaO\text{-}Al_2O_3\text{-}SiO_2\text{-}Fe_2O_3$ may dissolve MgO up to a maximum of 6 per cent. It is thereby assumed that these liquids containing MgO, and containing the same proportions of the four components, may exist in equilibrium with the same solid phases as the original liquids, at a somewhat lower temperature. For example, in the quaternary system a liquid of the composition 54·8 per cent. CaO, 22·7 per cent. Al_2O_3, 6 per cent. SiO_2, 16·5 per cent. Fe_2O_3 is at an invariant point and, at 1,338 deg. may exist in equilibrium with solid $3CaO \cdot SiO_2$, $2CaO \cdot SiO_2$, $3CaO \cdot Al_2O_3$ and $4CaO \cdot Al_2O_3 \cdot Fe_2O_3$. It is assumed that the liquid obtained by dissolving MgO in this liquid may exist in equilibrium with the same solid phases and that, when 6 per cent. MgO is present, it may also exist in equilibrium with MgO. These postulations are not strictly sound but, in connection with studies of the properties of Portland cement, it is assumed as a working hypothesis that they are approximately correct.

The equations of Group 3 are reproduced in Appendix I, the assumption being that the liquid remains in equilibrium with the crystallising phases until a temperature is reached at which all of the residual liquid solidifies as a glass. *G* represents the percentage of "quaternary glass." When magnesia is to be considered, the estimated percentage of glass, containing MgO, is not *G* but may be designated as *P*. Then, if *M* (designating MgO) is greater than $0{\cdot}06P$, $G = 0{\cdot}94P$ and free $MgO = M - 0{\cdot}06P$. If *M* is less than $0{\cdot}06P$, $G = P - M$ and free $MgO = O$.

A few calculations are shown in *Table* 3, obtained by the use of the above equations, which indicate probable changes in actual composition resulting from

arrested crystallisation. Eight commercial clinkers are represented (taken from the group listed in *Table* I) each of which had been reheated to about 1,400 deg. and cooled quickly with the object of producing relatively high glass content. Careful analyses were made, including free CaO, and the potential composition was calculated on the assumption of complete equilibrium crystallisation. These data are set down opposite the designation " potential " in the table. The glass content was approximated by the heat of solution method already described, and the compositions were recalculated to include glass on the assumption that normal crystallisation obtained to the estimated glass content. These values are set down under " calculated " in the table.

TABLE 3.—POTENTIAL COMPOSITION AT CRYSTALLINE EQUILIBRIUM AND CALCULATED COMPOSITION ASSUMING NORMAL CRYSTALLISATION TO AN APPROXIMATED GLASS CONTENT. ARRANGED IN ORDER OF INCREASING A/F RATIO.

No.	Condition	Type	Group	Series	A/F	Composition						Glass
						C_3S	C_2S	C_3A	C_4AF	MgO	Fr·CaO	
4Q	Potential	—	—	—	0.81	59.3	11.2	2.8	18.4	4.4	1.6	0
	Calculated	B	8b	45	—	55.8	10.6	0	4.6	3.1	1.6	22
11Q	Potential	—	—	—	0.95	67.0	13.7	3.5	13.2	0.8	0.3	0
	Calculated	B	9a	43	—	66.9	12.5	1.1	9.4	0.3	0.3	8
2Q	Potential	—	—	—	1.12	57.8	16.4	6.2	14.8	1.9	0.9	0
	Calculated	B	10b	44	—	59.4	11.1	0	5.0	0.6	0.9	21
8Q	Potential	—	—	—	1.65	52.0	26.1	7.9	9.0	3.5	0.8	0
	Calculated	A-1	2a	34	—	51.8	24.3	4.3	3.3	2.8	0.8	12
6Q	Potential	—	—		1.88	60.8	16.7	10.2	9.5	1.5	0.5	0
	Calculated	A-1	2a	34	—	60.6	14.6	6.0	2.9	0.7	0.5	14
3Q	Potential	—	—	—	2.60	51.6	25.6	10.3	6.0	4.0	0.4	0
	Calculated	A-1	2b	35	—	53.2	21.5	3.7	0	3.0	0.4	16
1Q	Potential	—	—	—	3.00	52.2	23.0	11.9	5.8	3.1	1.5	0
	Calculated	A-1	2a	34	—	52.1	21.8	9.5	2.0	2.6	1.5	8
7Q	Potential	—	—	—	3.47	51.2	22.2	14.7	6.0	3.5	0.6	0
	Calculated	A-1	2b	35	—	55.5	14.9	4.0	0	2.2	0.6	21

The same type of change is observed as in the calculations of Lea and Parker, but with the important difference that the glass recorded does not represent the total liquid content at 1,400 deg. but rather the amount of glass as actually determined. Experimental confirmation of some of these changes in constitution due to the presence of glass is given later.

Effect of Constitution on the Behaviour of the Cement.

Shortly after Rankin had defined the compounds of the CaO-Al_2O_3-SiO_2-system stable in the field of Portland cement, Bates[9] initiated the investigations at the National Bureau of Standards which have been continued up to the present time on the properties of the cement compounds and their effects on the cement.

A few examples will suffice to indicate important applications which have been made in the control of cement properties through a knowledge of clinker constitution.

A few years ago it was common practice to allow 21 days for the curing of a concrete road but information on the relative hardening rates of $3CaO . SiO_2$ and $2CaO . SiO_2$, together with finer grinding, has pointed the way to the manufacture of cements that permit the opening of roads in from two to seven days.

The building of large dams has necessitated the use of cements having low heat generation on hydration in order to reduce the thermal contractions on cooling which might induce serious cracking. These cements are designed to restrict the amounts of $3CaO . Al_2O_3$ and $3CaO . SiO_2$ in the clinker since these are the high heat-producing constituents.

Concrete exposed to natural or factory waters high in sodium sulphate or magnesium sulphate may be corroded owing to the formation of calcium sulphoaluminate. A vastly improved resistance is attained by the use of cements designed to limit the amount of calcium sulphoaluminate that may be formed.

Through similar applications of information on the behaviour of the compounds calculated to be present in clinker, cements have been improved in soundness, volume constancy, and durability, the rate of set has been more satisfactorily controlled, flash set and false set largely eliminated, dusting avoided, warehouse caking overcome and grindability of the clinker improved.

Of particular interest at the moment is the effect of the presence of glass, both directly and indirectly, on the properties of the cement. It has already been pointed out from theoretical reasoning that changes in the glass content are accompanied by changes in the amounts of crystalline constituents in certain directions. One of these changes involves periclase (free MgO), since MgO dissolves in the liquid to the extent of about 4 to 6 per cent.

GLASS CONTENT AND DELAYED UNSOUNDNESS DUE TO PERICLASE.—Microscopical observations have been made[21] on the clinkers listed in *Table* 1, one portion of each clinker being reheated and cooled quickly (*Q* sample), another portion similarly reheated and cooled slowly (*S* sample), and a third portion being the untreated plant clinker (*P* sample). Generally the amounts of periclase observed in polished sections (as measured with a Wentworth micrometer) have been found to be less than the total MgO of the clinker. This confirms the belief that some of the magnesia dissolves in the liquid. Furthermore, the average grain-size of periclase in the quickly-cooled clinkers, high in glass, was notably smaller than in the slowly cooled clinker, low in glass (compare *Plates* V and VI).

Further confirmation of the solution of MgO in the liquid was obtained through observation and examination of the residue obtained following the treatment of the clinker with nitric and hydrofluoric acids in the determination of heat of solution.[44] The residue was identified by Lerch as periclase. A systematic examination has revealed that, in the high-magnesia compositions, the slowly-cooled clinkers consistently showed a higher residue than the corresponding

quickly-cooled clinkers. The data for the clinker given in *Table* 1 are shown in *Fig.* 5 where the periclase remaining undissolved after ten minutes' treatment is plotted against the total MgO for both slowly and quickly-cooled clinkers. It may be observed that, with compositions containing more than 2 per cent. MgO, the periclase residue increases with increasing total MgO much more rapidly in the slowly-cooled than in the quickly-cooled clinkers. This may be interpreted as indicating either that less periclase is present in the quickly-cooled clinkers (the MgO being dissolved in the glass) or that it is present in a finer state of subdivision permitting more rapid solution in the acid mixture. The evidence given indicates that both conditions actually obtain.

It has been known for some time[48] that magnesia may give rise to excessive expansions in test specimens of neat cement stored in water for several years. Furthermore, it has been believed by some observers that a delayed unsoundness, attributable to MgO or a combination of MgO and free CaO, has been responsible for certain expansions in concrete road slabs and other structures.

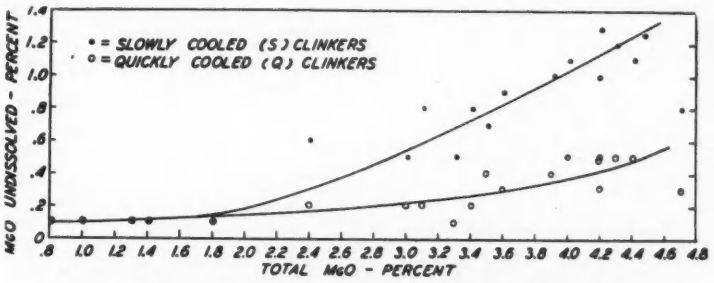

Fig. 5.—Relation between total MgO and the MgO remaining undissolved (420 g. 2N HNO_3 containing 5 ml. 48% HF) after 10 minutes.

An examination of a large number of commercial cements has been made by Lerch and Taylor at the Fellowship Laboratory[33]. Neat bars were prepared (1in. by 1in. by 6in.) with water to give normal consistency. After curing for one day in the moulds the bars were autoclaved for 72 hours at 177 deg. It was observed that the expansions were low when the MgO did not exceed 2·5 per cent. and free CaO 0·5 per cent. but that, with cements containing more than 2·5 per cent. MgO (low free CaO), some expanded greatly and others little. In other words, the MgO in one cement might cause serious expansion whereas the same amount of MgO in another cement might be without appreciable effect.

Clinkers were then prepared in a laboratory furnace and subjected to different heat treatments designed to result, on the one hand, in high glass content and, on the other hand, in low glass content. When these clinkers were examined for autoclave expansion it was observed that compositions containing 3 per cent.

[48] WHITE, A. H. *Proc. Am. Soc.*, Testing Materials, 1928, 28, 398.

MgO when slowly cooled (no glass) expanded seriously whereas the same compositions when cooled quickly (high glass) were scarcely affected.

This study was followed by the heat-treatment of the commercial clinkers referred to in Table 1. Tests on the cements prepared from these clinkers[49] showed that, with MgO content above 2·5 per cent., quick cooling gave cements of very low autoclave expansion, whereas by slow cooling the expansions increased with the MgO content. These data are shown graphically in *Fig.* 6. The conclusion appears that magnesia in the form of periclase is responsible for a delayed unsoundness whereas the same amount of magnesia dissolved in glass (or, in part, reduced in grain-size) does not bring about that condition.

A correlation between the autoclave expansion and the expansion of similar specimens in water and air over a long period has been made by Young[50]. Parallel

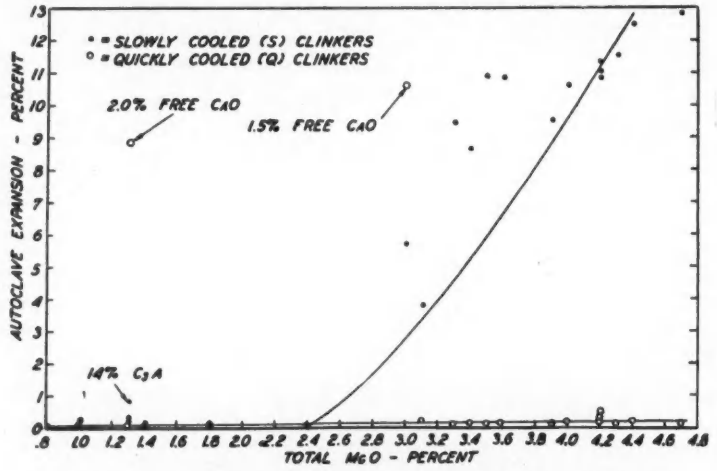
• = SLOWLY COOLED (S) CLINKERS
○ = QUICKLY COOLED (Q) CLINKERS
2.0% FREE CaO
1.5% FREE CaO
14% C3A
AUTOCLAVE EXPANSION - PERCENT
TOTAL MgO - PERCENT
0 1 2 3 4 5 6 7 8 9 10 11 12 13
.8 1.0 1.2 1.4 1.6 1.8 2.0 2.2 2.4 2.6 2.8 3.0 3.2 3.4 3.6 3.8 4.0 4.2 4.4 4.6 4.8

Fig. 6.—Relation between MgO Content and Autoclave Expansion. (5 hrs. at 215°C.)

tests were made in the autoclave, in water, and in air of 186 cements for periods ranging from four to nine years. Of these cements, 18 showed autoclave expansions above 1 per cent., and the water expansion of these 18 cements at four years was about 0·32 per cent. as compared with a water expansion averaging about 0·20 per cent. for the remaining 168 cements. At later ages the differences are more marked. In air storage (50 per cent. relative humidity) there is a distinct tendency for the cements showing delayed unsoundness first to contract for six to twelve months and then expand until in many cases disintegration occurs.

(To be continued.)

[49] Unpublished data from laboratory of Portland Cement Assoc., Chicago.

[50] YOUNG, R. N. *J. Am. Concrete Inst.*, 1937, 9, 13.

Refractories for Cement Kilns.

By HENRY G. FISK, Ph.D.

THE following abstract bibliography of the literature and patents from 1892 to 1938 relating to refractory linings for cement kilns has been prepared by Mr. Henry G. Fisk, Refractory Specialist of the Research Foundation of Armour Institute of Technology, Chicago, and formerly petrographer of the Universal Atlas Cement Co., and research chemist of the Calaveras Cement Co.

1892

(1) Kosman. Kiln lining for cement or lime kiln. *Tonind.-Ztg. 16*, 485–86, 21/5 (1892). (Wecke* 182).—The author suggests that, by using pure magnesia, clay containing masses can be prepared in a sufficiently refractory condition.

1899.

(2) W. Lessing and D. A. Wolff. Rotary kiln refractory. Ger. pat. 125,215 (1899), Brit. pat. 5923 (1900).

Tonind.-Ztg. 25, 2214, 7/12 (1901). (Wecke 184).—Air for combustion is forced through the pores of a refractory lining, thus preventing rapid burning out of the lining.

(3) E. H. Hurry and H. J. Seaman. Ger. pat. 150,845 (1899). (Wecke 185).—Process for producing a clinker coating on the lining of a rotary kiln. The lining is coated with a layer of salt or other suitable material which melts when heated, and when melted a layer of cement is caused to adhere to it and form a protective coating on the refractory.

1901

(4) Hemmoor and F. Valeur, Portland Cement Factory. Covering of a rotary kiln. Ger. pat. 134,820 (1901).

Tonind-Ztg. 27, 328, 28/2 (1903). (Wecke 186).—(1) Cement refractory with a granular clinker aggregate is used in place of broken stone or gravel. (2) Concrete with clinker aggregate is rammed or pressed into place in the kiln and there heated.

1903

(5) J. Jackson. Lining the rotary kiln. Brit. pat. 22,796 (1901).

Tonind.-Ztg. 27, 728, 25/4 (1903). (Wecke 187).—The kiln is lined with a mixture of ground brick and sodium silicate and other substances such as litharge, lime, etc., for the purpose of reducing waste of the refractory.

1904

(6) E. Riisager. Refractory kiln lining of cement concrete. *Tonind.-Ztg. 28*, 635, 7/5 (1904). (Wecke 187).—The author claims that the Valeur patent is not original.

(7) Anon. Refractory brick and refractory kiln linings. *Slate Trade Gazette*, No. 77, 101 (1904). (Wecke 188).—Dry Portland cement is mixed with 5 to 10 per cent. of water-free tar or tar residues and ground in a mill which is being heated. The resulting mass is moulded and burned. The bricks thus produced

* Abstract from Handbuch der Zementliterature, Friedrich Wecke (1927).

are black and hard and withstand considerable heat without melting or breaking. The plastic mass instead of being moulded can be placed directly on the hearth bed or walls.

1907

(8) C. Löser. A contribution to the working of cement mixes on refractories. *Protokoll 30*, 191–99, 21/2 (1907). (Wecke 191).—The author conducted tests in a recuperative furnace from which it is concluded that by properly processing a pure clay the surface of the refractory can be protected against corrosion.

(9) Chemical Laboratory for the Clay Industry. Dynamidon (high-alumina cement kiln refractory). *Tonind.-Ztg. 31*, 1412–13, 5/9 (1907). (Wecke 191).—Heating tests showed that at Seger cone 39 no appreciable alteration of the brick took place.

(10) J. Schutt. Mortar for laying up the lining of kilns for burning cement or the like. Ger. pat. 220,383 (1907). (Wecke 192).—If Portland cement mortar is used it is improved by the addition of hydrated lime.

1908

(11) Anon. The effect of cement on refractory linings. *Cement Eng. News. 20*–35 ; *C.A. 2*, 1611 (1908).—Experiments of Dr. Löser, of Halb, show that alumina resists the action of cement clinker well at high temperatures but clay mixture containing silica does not. Therefore, it appears that a comparatively small proportion of silica in the lining of a cement kiln is a serious disadvantage.

1910

(12) Valeur. Gummden, Austria. Concrete from neat cement as a material for refractory lining of cement kilns. *Z. angew. chem. 23*, 1458–60 ; *Chem. Abs. 4*, 2991 (1910) ; *Concrete & Constr. Eng. 3*, 476–7. (Wecke 193). The author employed successfully for lining rotary and shaft kilns a " concrete " prepared from a mixture of equal parts by weight of cement clinker (2–20 mm. diameter) and ground slow-setting cement, with just sufficient water to make the mass plastic. This material was found to preserve its form and hardness upon either slow or rapid heating to temperatures as high as 1770 deg. F. It is unacted upon by the charge in the kiln except for slight mechanical erosion, and there is no tendency for accretions to form. Such a lining 20 cm. thick will last from 3 to 6 months in the sinter zone and for some years in the rest of the kiln. The cost of installation and repairs is far less than ordinary firebrick lining.

(13) J. H. Schutt. Process for preparing refractory brick or kiln lining. *Ger. Anm. Sch. 33*, 662 ; kl 80B (1909).

Ausgel. 17/3 (1910). (Wecke 193).—Cement granules, cement and lime, or magnesia, or dolomite are mixed with water and moulded, or pressed into the kiln.

1911

(14) J. H. Schutt. Mortar for laying up the lining of cement kilns or the like. Ger. pat. 243,933. (Wecke 195).—(1) Cement granules omitted in Patent No. 220,383 ; (2) Preparation of the kiln lining by using mortar as a rammed mass ; (3) Preparation of bricks from mortar which are laid up as a kiln lining and burned in place.

(*To be continued.*)

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